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ABSTRACT

Basic knowledge about Skylab experiments is presented in this book, one of a series, for the purpose of informing high school teachers about scientific research performed in orbit and enabling the teachers to broaden their basis for material selection. This third volume is concerned with the effect of a weightless environment on melting and resolidification of metals and semiconductor crystals and on combustion of solid flammable materials. The first section provides an introduction to crystal growth and a unifying background to the experiments. The second section is related to experiments on gallium arsenide crystal growth, vapor growth of IV-VI compounds, immiscible alloy compositions, radioactive tracer diffusion, microsegregation in germanium, growth of spherical crystal, whisker and reinforced composites, indirum antimonide crystals, mixed III-V crystal growth, halide eutectecs, silver grids melted in space, aluminum-copper eutectic, and classrooom demonstrations. The third section discusses zero gravity flammability, metals melting experiment, and exothermic brazing experiments. A description of the Materials Processing Facility is given in the fourth section. Materials are suggested to serve as an aid in developing future curriculum supplement materials. Included are a glossary and a bibliography. (CC)

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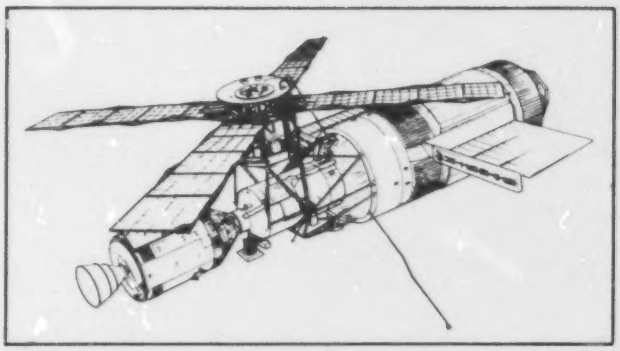
Skylab Experiments

Volume 3
Materials Science



Information for Teachers, Including Suggestions on Relevance to School Curricula.

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

SE 017 788

Skylab Experiments

Volume 3
Materials Science

Produced by the Skylab Program and NASA's Education Programs Division in Cooperation with the University of Colorado

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Washington, D.C. 20546, May 1973

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PREFACE

Characteristically, new scientific knowledge reaches general application in classrooms years after it has been obtained. This long delay stems, to a large extent, from a lack of awareness that information is available and that it has relevance to secondary school curricula. To accelerate this process, the National Aeronautics and Space Administration has prepared a series of documents concerning Skylab experiments to apprise the educational community in detail of the investigations being conducted in the Skylab Program, and the types of information being produced.

The objective is not to introduce the Skylab Program as a subject in the classroom, but rather to make certain that the educational community is aware of the information being generated and that it will be available for use. Readers are urged to use these books as an aid in planning development of future curriculum supplement material to make the most appropriate use of this source of scientific knowledge.

National Aeronautics and Space Administration
Washington, D.C. 20546
May 1973

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INTRODUCTION

The Skylab Education Program

This year the United States' first manned scientific space station, Skylab, was launched into orbit to be the facility in which successive crews of astronauts can perform more than 270 scientific investigations in a variety of fields of interest. These investigations can be divided into four categories: physical sciences, biomedical sciences, earth applications, and space applications.

The Skylab Program will produce information that will enhance present scientific knowledge and perhaps extend the frontiers of knowledge on subjects ranging from the nature of the universe to the structure of the single human cell. It is the objective of the National Aeronautics and Space Administration that the knowledge derived from the Skylab Program's investigations be made available to the educational community for applications to high school education at the earliest possible date.

For this reason, the Skylab Education Program was created to assure that maximum educational benefits are obtained from the Skylab effort, documentation of Skylab activities is adequately conducted, and understanding of scientific developments is enhanced.

This document, one of several volumes prepared as part of the Skylab Education Program, has the dual purpose of (1) informing high school teachers about the scientific investigations performed in Skylab, and (2) enabling teachers to evaluate the educational benefits the Skylab Program can provide.

These books will define the objectives of each experiment, describe the scientific background on which the experiment is based, outline the experimental procedures, and indicate the types of data anticipated.

In preparing these documents an attempt has been made to illustrate relationships between the planned Skylab investigations and high school science topics. Concepts for classroom activities have been included that use specific elements of Skylab science as focal points for demonstrations of selected subjects. In some areas these address current curriculum topics by providing practical applications of relatively familiar, but sometimes abstract principles; in other areas the goal is to provide an introduction to phenomena rarely addressed in high school science curricula.

It is the hope of the National Aeronautics and Space Administration that these volumes will assist the high school teacher in recognizing the educational value of the information resulting from the Skylab Program which is available to all who desire to make use of it.

Application

Readers are asked to evaluate the investigations described herein in terms of the scientific subjects taught in secondary schools. The related curriculum topics identified should serve as suggestions for the application of Skylab Program-generated information to classroom activities. As information becomes available from the Skylab Program, announcements will be distributed to members of the educational community on the NASA Educational Programs Division mailing list. To obtain these announcements send name, title, and full school mailing list (including zip code) to:

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Washington, D.C. 20546
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This volume deals with the materials science and technology investigations conducted on Skylab. The thirteen experiments that support these investigations have been planned to evaluate the effect of a weightless environment on melting and resolidification of a variety of metals and semiconductor crystals, and on combustion of solid flammable materials. The first section of the book serves as an introduction to solidification and crystal growth and provides a unifying background to the experiments. Section 2 discusses experiments related to crystal growth and solidification of metals. Section 3 discusses experiments in welding and brazing, and flammability; and Section 4 contains a description of the materials science experiment facilities on Skylab. Appendix A contains a glossary and Appendix B a bibliography.

Attempts have been made to identify relationships between the Skylab science and classroom science curricula. These are discussed in Sections 1, 2 and 3 and are summarized in Table 1.

Table 1 Related Curriculum Topics

SECTION 2 PHYSICAL METALLURGY & CRYSTAL MANUFACTURE (M553, M555, M556, M557, M553, M559, M560, M561, M562, M563, M564, M565, M566)	SECTION 3 SPACE OPERATIONS - SUPPORT EXPERIMENTS (M479, M551, M552)
CHEMISTRY	
Composition, crystal growth, crystal structure alloys, electrical conductivity, IV-VI compounds, III-V compounds, phase diagrams, whisker growth, supercooling, semiconductors, properties of materials in vacuum, surface tension, radioactive tracer techniques, zone refining, ultrafiltration porosity, flame tests, and spectroscopy	Combustion, explosive mixture ranges, composition, oxidation-reduction, alloys, flash point and flammability, phase changes, convection and buoyancy, specific heat, electron microscopy, adhesion, and cohesion
INDUSTRIAL ARTS	
Finishes, metal forming, reinforced materials	Welding, brazing
METALLURGY	
Structure, nucleation crystals, alloys, tensile strength, ductility, malleability, sphericity, zone refining	Microstructure alloys
PHYSICS	
Heat, vacuum, electrical properties, zero gravity, strength of materials, semiconductors, phase changes, droplet formation, surface tension, radioactive tracer techniques	Convection, heat transfer, ignition and flash point buoyancy, diffusion, electron microscopy, surface properties, adhesion and cohesion, heat of fusion, and vaporization
GENERAL SCIENCE	
Crystal growth, flame tests, phase changes, diffusion	Kindling temperature, combustion, flame extinguishing, flame proofing, dust explosion demonstrations: explosive mixture range, heat transfer, convection currents, crystal growth, purification by crystallization

Acknowledgments

Valuable guidance was provided in the area of relevance to high school curricula by Dr. James R. Wailes, Professor of Science Education, School of Education, University of Colorado; assisted by Mr. Kenneth C. Jacknicke, Research Associate on leave from the

University of Alberta, Edmonton, Alberta, Canada; Mr Russel Yeany, Jr., Research Associate, on leave from the Armstrong School District, Pennsylvania; and Dr. Harry Herzer and Mr. Duane Houston, Education and Research Foundation, Oklahoma State University.

The Skylab Program

The Skylab orbiting space station will serve as a workshop and living quarters for astronauts as they perform investigations in the following broad categories: physical sciences, biomedical sciences, Earth applications, and space applications.

The spacecraft will remain operational for an eight-month period, manned on three occasions and unmanned during intervening periods of operation. Each manned flight will have a crew of three different astronauts. The three flights are planned for durations of one month, two months, and two months, respectively.

A summary of objectives of each of the categories of investigation follows.

Physical Science

Observations free of filtering and obscuring effects of the Earth's atmosphere will be performed to increase man's knowledge of (1) the sun and of its importance to Earth and mankind, and (2) the radiation and particulate environment in near-Earth space and the sources from which these phenomena emanate.

Biomedical Science

Observations under conditions different from those on Earth will be made to increase man's knowledge of the biological functions of living organisms, and of the capabilities of man to live and work for prolonged periods in the orbital environment.

Earth Applications

Techniques will be developed for observing from space and interpreting (1) Earth phenomena in the areas of agriculture, forestry, geology, geography, air and water pollution, land use and meteorology, and (2) the influence of man on these elements.

Space Applications

Techniques for adapting to and using the unique properties of space flight will be developed.

The Skylab Spacecraft

The Skylab cluster contains five modules (see illustration).

1) The *orbital workshop* is the prime living and working area for the Skylab crews. It contains living and sleeping quarters, food preparation and eating areas, and personal hygiene equipment. It also contains the equipment for the biomedical science experiments and for some of the physical science and space applications experiments. Solar arrays for generation of electrical power are mounted outside this module.

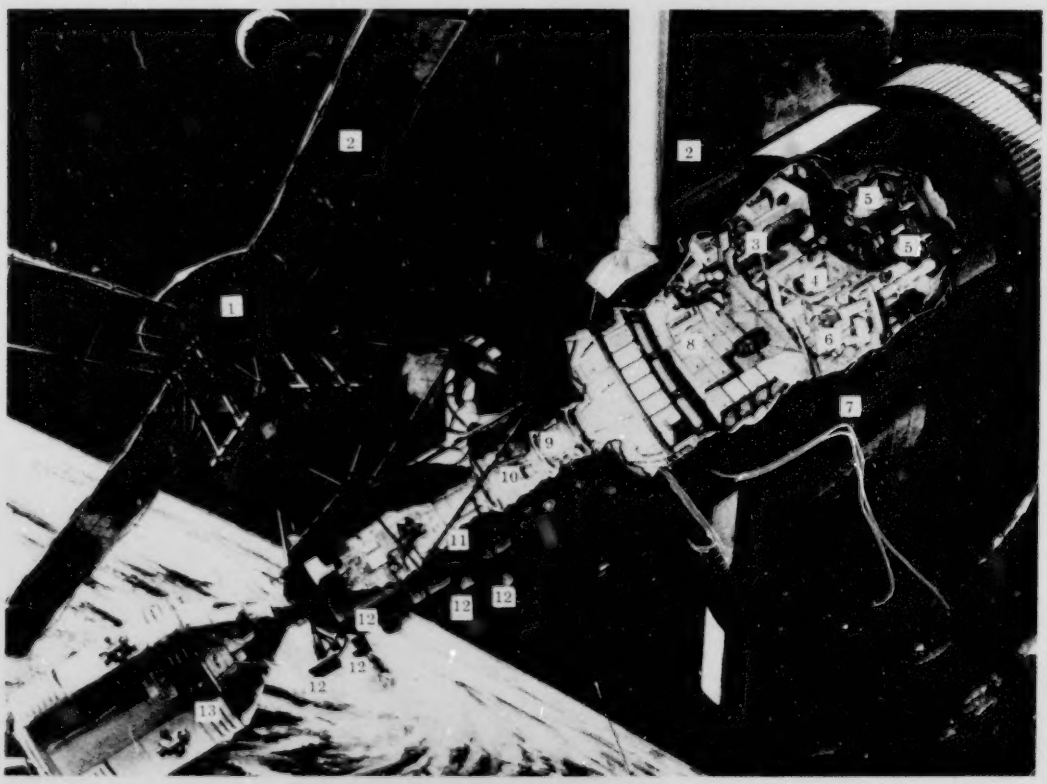
2) The *airlock module* contains the airlock through which suited astronauts emerge to perform activities outside the cluster. It also contains equipment used to control the cluster's internal environment and the workshop electrical power and communications systems.

3) The *multiple docking adapter* provides the docking port for the arriving and departing command and service modules, and contains the control center for the telescope mount experiments and systems. It also houses the Earth applications experiments and materials science and technology experiments.

4) The *Apollo telescope mount* houses a sophisticated solar observatory having eight telescopes observing varying wavelengths from visible, through near and far ultraviolet, to X-ray. It contains the gyroscopes and computers by which the flight attitude of Skylab is controlled. Solar arrays mounted on this module generate about half of the electrical power available to the cluster.

5) The *command and service module* is the vehicle in which the crew travels from Earth to Skylab and back to Earth, and in which supplies are conveyed to Skylab, and experiment specimens and film are returned to Earth.

Skylab will fly in a circular orbit about 436 kilometers (235 nautical miles) above the surface of the Earth, and is planned to pass over any given point within latitudes 50° north and 50° south of the equator every five days. In its orbital configuration, Skylab will weigh over 91,000 kilograms (200,000 pounds) and will contain nearly 370 cubic meters (13,000 cubic feet) for work and living space (about the size of a three bedroom house).



1 Apollo telescope mount
2 Solar arrays
3 Sleeping quarters
4 Personal hygiene
5 Biomedical science experiment
6 Ward room
7 Orbital workshop
8 Experiment compartment
9 Airlock module
10 Airlock external hatch
11 Multiple docking adapter
12 Earth resources experiments
13 Command and service module

Skylab Orbiting Station

Section 1

Introduction

BACKGROUND

Since the Industrial Revolution, progress made in metals development has undergone significant acceleration. For example, in the past 20 years, improvements in alloying have almost doubled the strength of steels and other structural metals. Research in other fields has improved other physical properties such as corrosion resistance, thermal and electrical conductivity, stability, workability, and tolerance to extreme temperatures, and has brought "new" metals such as titanium into commercial usage.

The processing of metals is influenced by the Earth's environmental characteristics such as the atmosphere in which the processing occurs and gravity. The former can introduce adverse contaminating influences, but these can be minimized by use of artificial atmospheres such as inert gases. Nothing practical can be done about gravitational effects, at least not on Earth. These gravitational effects include separation of liquid metals of different densities, uneven cooling characteristics and possible chemical contamination from the vessel in which the metal must be contained.

These undesirable gravity-related effects may be minimized in the weightless environment of orbital flight where differences in density have little significance and solidification without contact with a container is possible. Within the orbiting Skylab, experiments will be performed to provide research data with respect to the effect of weightlessness on metal processing.

A few of the basic principles of metallurgy are presented as background information in support of the processes involved in the experiments. For more detailed data, refer to the Bibliography.

MATERIALS

The Skylab materials science and technology investigations deal mainly with metallic materials. The metals studied include the familiar steel, aluminum, copper, nickel, and silver and the less familiar gallium, germanium, indium, and tellurium which may not be commonly recognized as metals:

The shaded area of the periodic table shown in Figure 1-1 identifies those elements that have the metallic characteristics, including those elements in the borderline region that have aspects of both metals and nonmetals.

CHARACTERISTICS OF METALS

Most substances that are classified as metals have common physical characteristics. They are generally light in color and

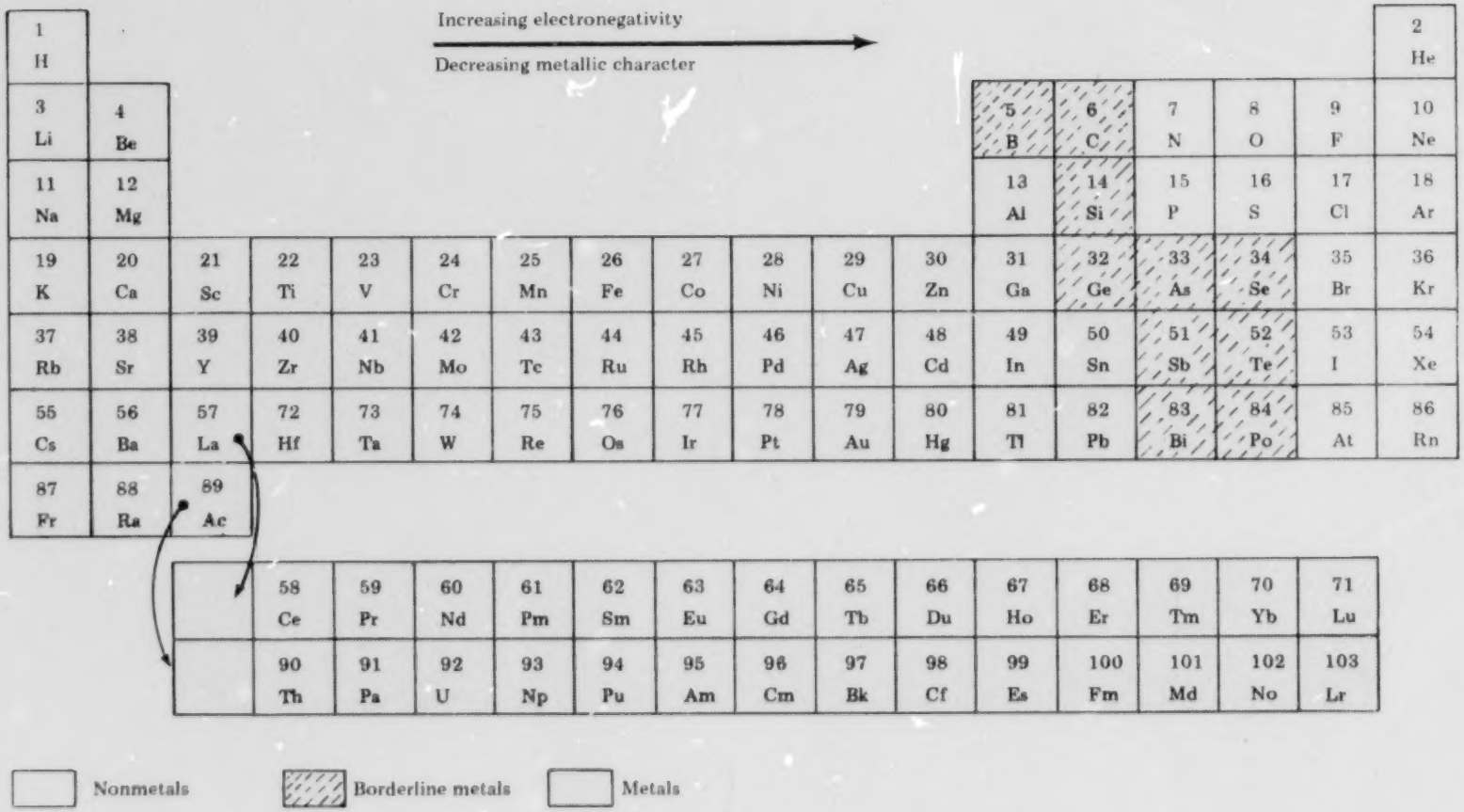


Figure 1-1 Periodic Table

have a lustrous appearance; they have high tensile strength, elasticity, and ductility; they combine with oxygen to form oxides; they have electrical and thermal conductivity; and they may be manipulated by machining, forming, and extrusion. The different metals in their pure state have these characteristics in varying combinations and degrees; i.e., copper has high electrical and thermal conductivity, but low tensile strength; iron has high tensile strength, but low conductivity.

Change in Characteristics of Metal

The characteristics of a metal can be selectively changed by the addition of other elements. This combination of other chemical elements is called an alloy. Some of the variations in physical properties obtainable by alloying are (1) the addition of less then 1% carbon to iron produces steel having greatly increased tensile strength; (2) the introduction of a small quantity of vanadium to a steel alloy increases the hardness potential so that it can be used for cutting other steel alloys; and (3) adding 2% thorium oxide to nickel produces a marked increase in high temperature strength.

Many important alloy combinations have properties which could not be predicted on the basis of the properties of the constituent metals. For example, copper and nickel, both having good electrical conductivity, form alloys having very low conductivity, or high resistivity, making them useful as electrical resistance wire.

SEMICONDUCTORS

A semiconductor is a material that has electrical conductivity characteristics that range between that of a good conductor and an insulator. Pure silicon and germanium function as semiconductors because the electrons in these crystals are not as free to move as in good conductors such as copper or aluminum. In order to make the semiconductors useful as transistors, small traces of another element are added to the crystal to provide the excess of positive or negative charges necessary for a specific degree of conduction. The process of adding the substance is called doping the crystal and the substance added is called the dopant. The addition of the dopant enhances conductivity of the semiconductor crystal; i.e., if boron is added to pure silicon at the ratio of one part boron per million parts silicon, the conductivity is increased by a factor of about one hundred thousand.

Dopant—a small quantity of a substance added to another substance to alter the latter's properties

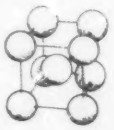
Electronic circuitry today is made mostly with semiconductor material. The state of the art in semiconductor development has evolved to the point where microminiature circuits are now widely used. A typical silicon semiconductor chip that measures only a few millimeters across can incorporate 200 devices (such as transistors and diodes) on its surface to perform a complex function, such as running a pocket calculator.

The density of 200 devices on a single chip could be increased about a hundred times if a perfect semiconductor crystal could be manufactured. On a piece of silicon less than one inch in diameter, 750,000 devices could be incorporated to serve as a photocathode of a television camera capable of resolution of images with extremely low level light sources. This is not possible yet because of manufacturing limitations.

SOLIDIFICATION OF METALS

Structure

Pure Metals—The atoms in a metal, in the liquid phase, move freely, but as the temperature is lowered they lose energy and their motion becomes increasingly sluggish. At the freezing temperature, or slightly above, nuclei (small clusters of atoms in a definite crystalline arrangement) begin to form. Because of internal temperature variations some nuclei melt back to the liquid phase while in other locations in the melt new nuclei begin to form. As the temperature decreases, the rate of formation of nuclei exceeds that of melting and the crystals grow larger.



Body centered cubic

Face centered cubic

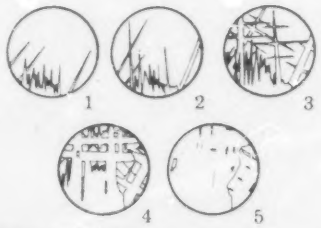
Hexagonal close packed structure

As solidification proceeds, the crystal nuclei form in different directions and the atoms tend to become disposed in a definite geometrical pattern known as the space lattice.

The particular basic geometric arrangement of atoms within the crystal depends on the particular metal. In general, metal crystals are arranged in one of three patterns. There are the body-centered cubic, the face-centered cubic, and the hexagonal close packed structures. This orderly structure repeats its basic form in geometric cells aligning their parallel facets throughout the crystal.

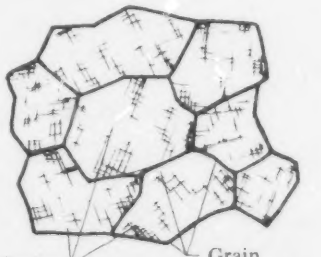
Dendrites

The crystals attract other atoms to their space lattice and develop crystalline structures called dendrites. If heat is continually removed from the solid to liquid interface, the dendrites will expand and grow into the liquid metal. This growth progresses until a neighboring crystal of the same form but of a different orientation obstructs the growth. Each of the resulting crystals is called a grain and the interface between the grains is called the grain boundary.

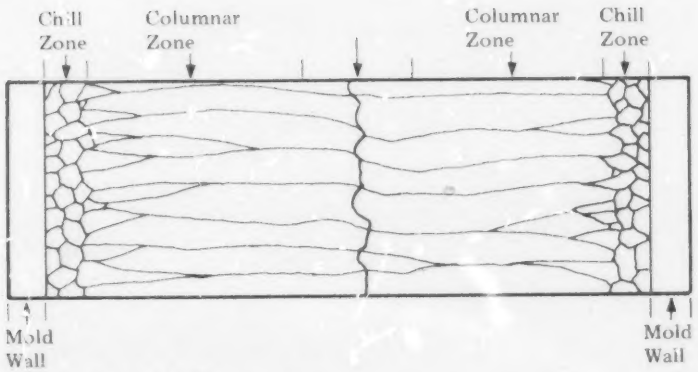


Dendritic Growth

If the temperature is maintained just below the freezing point, the rate of nucleation is low and fewer crystal growth sites develop. The resulting crystals grow larger because there are fewer neighboring crystals to obstruct the growth. If the temperature is lowered in such a way that no nuclei are formed at the freezing point, or below, the metal is said to be undercooled. There is a low temperature in an undercooled metal where nucleation sites occur uniformly and rapidly throughout the melt and the metal freezes with many small sized grains.



Grains (Crystals)



Section View of a Casting

The sketch illustrates the crystal growth sizes in a typical metal casting. The outer edges have small grains because the walls of the container tended to undercool that region or impurities in the container acted as nucleation sites. Further into the casting, the rate of nucleation was lower and fewer nuclei were present to interfere with the growing crystals.

Nucleation—to bring together into a nucleus

The larger crystal grains in a casting result in a weaker overall structure because tiny cleavage cracks will tend to progress rapidly along the facets in aligned crystal form. Conversely, multioriented crystals of many small grains tend to impede the advance of a cleavage crack. It is important then, from the standpoint of strength, that a casting be made of many small grains.

Solidification Temperatures

Alloys—The freezing of an alloy follows the same crystal growth sequence as a pure metal; however, as the constituent elements may have different solidification temperatures, the change from liquid phase to solid phase occurs over a temperature range. Within this range both phases can exist for both materials. A graphic representation of this temperature range is shown in Figure 1-2.

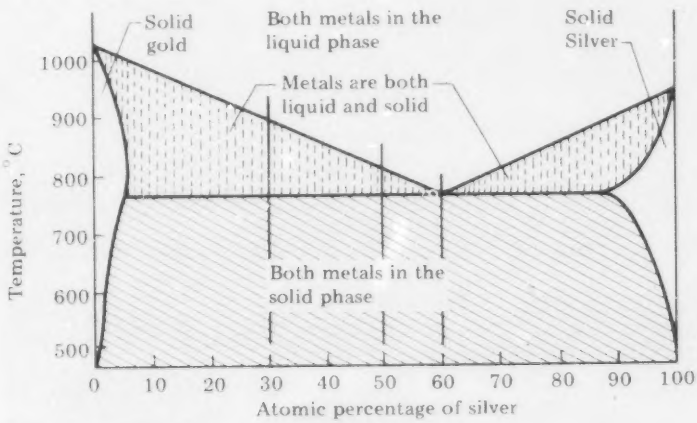


Figure 1-2 Phase Diagram of Silver and Gold

Figure 1-2 Photomicrograph of TD Nickel

Temperature Range

This figure shows the variation of the dual phase temperature range with variation of composition of the alloy. It will be seen that as the percentage of silver in the gold/silver alloy is increased toward 60%, the dual phase temperature range diminishes. At the 60% silver point the temperature range is zero, i.e., above the temperature shown (779°) both metals are liquid and below 779° both are solid. This composition is called the eutectic composition.

Detailed analysis of an alloy having the eutectic (60% silver, 40% gold) composition would show a homogeneous structure with the proportions of each element being uniform throughout the melt. In another alloy, for example 30% silver, 70% gold, the percentage composition would vary throughout this solid with silver-rich particles being contained in a gold-rich matrix. This is caused by the freezing solvent (gold) rejecting some of the liquid solute (silver). The process is called segregation and the result is undesirable in an alloy, from the standpoint of maintaining homogeneous characteristics such as corrosion resistance.

FORCES INFLUENCING SOLIDIFICATION

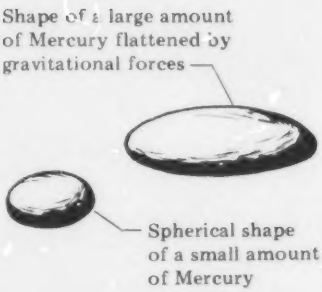
The forces that have an effect on the composition or shape of a solidified metal are volume changes, surface tension, and gravity induced segregation and convection.

In producing an alloy with uniform distribution of elements, it is important to insure that the temperature during the molten phase is uniform throughout. As a metal is starting to solidify, however, quantities of hot liquid are in motion within the melt. The hot less-dense liquid tends to rise causing a turbulent thermal condition. Because of this, there is no uniform temperature distribution and the alloyed metal solidifies with an uneven distribution of elements.

An alloy is also affected by gravity-induced segregation. The less-dense material tends to float upward in the molten phase causing a nonuniform distribution of elements. These two factors are presented as problems in obtaining a good, uniformly composed alloy. In general, these problems are apparent in the processing of all metals but for most structural or decorative use of metals it is not really necessary to achieve the ideal (perfectly uniform) distribution of elements. Large metal structures exhibit their particular alloy characteristics just by the average distribution of elements. However, in very small structures of metal such as semiconductor crystals or the tiny wires used in a heart pacemaker, the uniformity of composition becomes very important.

Surface tension is the force that holds a small drop of liquid on a flat surface in the shape of a flattened sphere. As the amount of liquid (i.e., the mass) is increased, the gravitational

force on the liquid overrides the surface tension and the liquid spreads to assume a much flatter shape. The profile of the edge of the "pool" continues to reflect the profile of the original drop (i.e., the effect of the surface tension is evident) but the original nearly spherical form is lost. In a weightless environment the surface tension is expected to become the controlling force regardless of the mass of the liquid.



Liquid to Solid

A change in volume characteristically accompanies a change of state from liquid to solid. Usually this change is a reduction in volume. In a few special cases the volume increases, such as freezing water to ice, a common example, and gallium which increases in volume as it solidifies.

Liquids allowed to solidify on Earth generally have a form as shown in Figure 1-3. The hollow (or pipe) on the top of the ingot results from the flow of the remaining liquid into the spaces between the already solidified dendrites. Solidification of a liquid in a weightless environment is expected to result in the formation of a cavity (or cavities) inside the spherical body with no apparent change in outside shape. High internal stresses will result from the combination of volume change due to solidification and the thermal stresses of cooling.

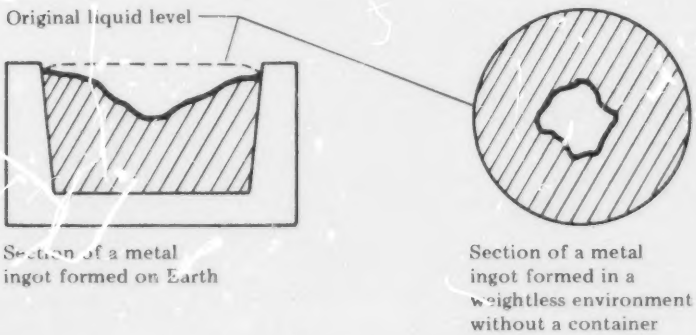

Original liquid level
Section of a metal ingot formed on Earth
Section of a metal ingot formed in a weightless environment without a container

Figure 1-3 Volume Change in a Metal

THE ROLE OF CONVECTION IN COMBUSTION

Convective flow, discussed earlier as a disruptive influence in the formation of homogeneous solids, has a major role in the much more easily appreciated process of combustion. It is convection that replenishes the oxygen needed to sustain combustion. In the case of severe forest fires, the inrush of air powered by convective flow produces local high winds that make the situation worse. In a weightless environment, gravity-induced convection should not occur and the combustion process will be different. This condition will permit detailed study of other aspects of combustion that are usually masked by the convection effects. These include heat gradients, flashover, and self-extinguishment.

SKYLAB MATERIALS SCIENCE INVESTIGATIONS

Sixteen experiments are planned to be performed on Skylab to study the aspects of materials science briefly discussed in the preceding paragraphs. The experiments are:

M553—Sphere Forming

M555—Single Crystal Growth

M556—Vapor Growth of IV-VI Compounds

M557—Immiscible Alloy Compositions

M558—Radioactive Tracer Diffusion

M559—Microsegregation in Germanium

M560—Growth of Spherical Crystals

M561—Whisker-Reinforced Composites

M562—Indium Antimonide Crystals

M563—Mixed III-V Crystal Growth

M564—Halide Eutectics

M565—Silver Grids Melted in Space

M566—Aluminum-Copper Eutectic

These thirteen experiments are described in Section 2. The following three experiments are described in Section 3:

M479—Zero Gravity Flammability

M551—Metals Melting

M522—Exothermic Brazing

CREW ACTIVITIES

A Skylab crew member will follow prescribed procedures while performing the materials experiments. He will prepare the work areas for each experiment, obtain the particular experiment specimen and install it in the appropriate facility, and initiate the experiment process. He will observe and record any outward reactions that the experiments produce. He will control the heating and cooling rates used in some experiments, vent or depressurize and repressurize the work chamber as appropriate, and provide water sprays when required.

Finally, he will remove and store the experiment specimens and apparatus and clean the work areas in preparation for the next experiment.

DATA

Information resulting from these experiments will be derived from postmission analysis of telemetered data and returned specimens formed.

Photographic data of those activities suitably exposed will also be available. The specific data generated by each experiment will be discussed in that experiment description. Generally this data and the results of specimen analyses are expected to be available between 90 days and a year after performance of the experiment.

RELATED CLASSROOM DEMONSTRATIONS

Water may be used to demonstrate some of the solidification processes of metals.

Crystal Growth

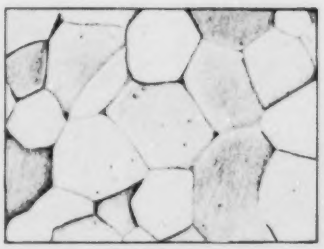
- Nucleation and crystal growth can be demonstrated with water by lowering the temperature of pure water below its freezing point. Foreign objects of small radii should not be present in the water, and it is sometimes necessary to wax the container. At a temperature of about -4° or lower, drop a small ice crystal into the undercooled water and it will freeze immediately. The ice crystal acted as a nucleation site.

Dendrite Growth

- Dendritic growth of ice crystals can be observed by lowering the temperature of a glass plate to below the freezing point of water (i.e., by chilling the plate in the freezer section of the refrigerator), and maintaining that temperature while allowing cool water vapor to condense on the plate. This produces frost patterns that are characteristic of dendritic growth. (See illustration.)

Dendrite growth of water crystals

- The grain boundaries of a piece of metal are normally invisible. A metallographic technique to observe a metal specimen is to etch the grains to make them visible. The metal specimen may be prepared by highly polishing and etching it with a dilute etchant and washing it with alcohol and water. The specimen may then be observed using a microscope with low angle illumination. (It may require a second polishing and etching to produce a useful sample, as illustrated.) The etching fluids are usually dilute (2-4%) alcholic solutions of acids or bases. Examples include:
  - steel, 2% nitric acid in alcohol or 4% picric acid in alcohol;
  - gold and platinum, aqua regia;



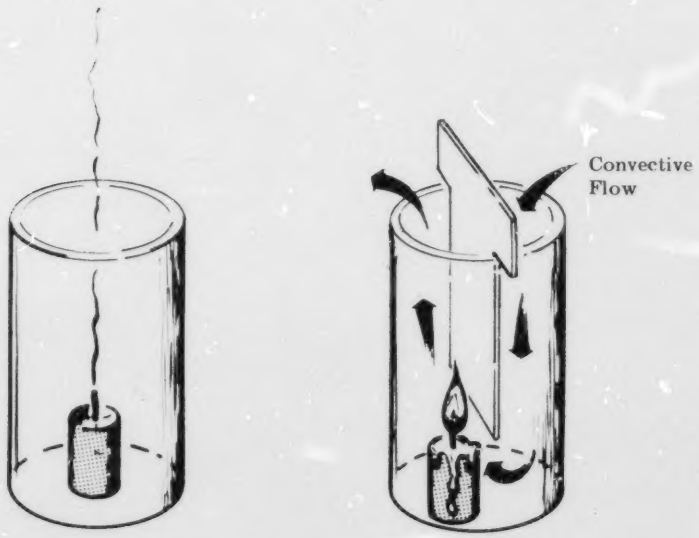
Microscopic view of grain boundaries made visible by chemical etching

- brasses and copper alloys, -2% soiution of ferric chloride;
- tin, 2% nitric acid in alcohol;
- aluminum, hydrofluoric acid.

- Look closely at a piece of galvanized steel. Note the fan-like groupings of zinc crystals covering the surface. These are grains of zinc crystals overlapping each other.

Convection

- The role of convection in combustion is easily demonstrated by placing a candle at the base of a container, such as the chimney of a kerosene lamp. The base of this chimney is closed and the top is open. Note how long the candle continues to burn. Now suspend a T-shaped piece of material reaching almost down to the flame and slightly offset. The air mass begins a convective flow down one side of the divider and up the other side.



Convection in Combustion

Section 2

Physical Metallurgy and Crystal Manufacture

This section describes a series of experiments designed to investigate the physical, chemical, and thermal mechanisms that control the manufacture of a variety of metallic materials. By performing these investigations in the weightless environment of Skylab, information can be derived related to the current theories about the influence of gravity on the physical and chemical processes of melting and solidification of metals.

BACKGROUND

Crystal Formation

The investigations discussed in the following paragraphs deal with the effect of gravity on crystal formation, on the mixing of metals in alloys, and on the value of surface tension as a shaping influence.

In Section 1 the effect of uneven heat flow on the crystalline structure of metals was discussed. Variations in crystal size occur because of uneven conditions of undercooling arising from heat loss to the atmosphere surrounding the melt or to the container in which the metal solidifies. In this weightless environment, small quantities of pure metals and alloys solidified in a free floating condition out of contact with external heat sinks will be compared with identical melts that solidified while still attached to a support.

The alloy combinations used in these investigations include materials of different melting temperatures and densities. Solidification of alloys having components with such differences can result in segregation in which the central portion of a dendritic crystal is richer in the element having the higher freezing point. If the lower freezing point also has the lower density, the segregation can also be accompanied by stratification of the constituent elements when the process is performed on Earth. This phenomenon should not occur in the weightless environment of Skylab.

Use of metallic mixtures with wide density differences results in undesirable stratification on Earth to the extent that the properties sought cannot be achieved. An example is the possible use of crystal "whiskers" of very high tensile strength to reinforce low strength metals.

Whiskers—Elongated single crystals can be used to reinforce low strength metals.

By uniformly distributing these whiskers through a lower strength material, a composite material of very high strength can be achieved. Relative densities play a significant role in whisker reinforced materials. In these cases the density differences cause the whiskers to float on the surface of composite material. In a gravity-free environment, uniform distribution should be possible.

Another gravity-related phenomenon that influences metal solidification is convection while the material is in the liquid

phase. Convective flow of the liquid metal can occur in the whole melt or in separate areas depending on variations in local temperatures. These flows can have adverse influences on the resulting metal properties, particularly in the area of disrupting segregation.

Depending on the required properties, segregation of constituent elements in the metal mixtures may or may not be desirable. Solidification of certain metal mixtures of eutectic composition in which the freezing temperatures of the elements are the same, results in distinct segregation of the crystals of the components. Rejection of one component by the other occurs within the liquid phase just ahead of the crystal growth and can cause parallel crystal formation in which lamella, or rods, develop through the solid.

Lamellar Crystals

These linear or lamellar crystals can introduce desirable properties such as super conduction of electricity, optical characteristics, directional magnetism, etc. Local or general convective flows can prevent the formation of these structural features by disrupting the development of the crystals.

Lamellar—parallel plates of metal crystals resulting from a single melt.

With the elimination of gravity-related stratification or convective flow, other forces become influential in material processing. These include diffusion and surface tension. Diffusion of one material through another occurs by the probabilistic migration of the atoms. The paths of the individual atoms is random but there is a general flow from areas of high concentration to areas of low concentration. Diffusive flow is generally accelerated by heating, i.e., the motion of the atoms is more energetic. On Earth, gravity can play an important part especially when a significant density difference occurs. In a weightless environment, the gravitational influence is eliminated and the diffusion can be studied.

The relative roles of gravity and surface tension in forming metals in the liquid phase was mentioned in Section 1. The influence of surface tension on molten metals will be studied in two investigations in the Skylab environment. A homogeneous material in the liquid phase in free fall should form a sphere and since no container is required to hold it in space, it will retain that shape while solidifying.

The experiments related to the above areas of investigation, and described in the following paragraphs, are all performed within the Skylab Materials Processing Facility. This equipment is described in Section 4 of this volume.

Except for experiments M553 and M555, the crystal manufacturing experiments in this section will be performed in similar procedures. The metal specimens for each

experiment are encased in cartridges (Figure 2-1) of similar configuration that fit in the electric furnace. There are individual differences between the cartridges and the furnaces in the arrangement of heat flow paths. The experiment specimen melting and freezing takes place entirely inside the cartridge.

Experiment Cartridge

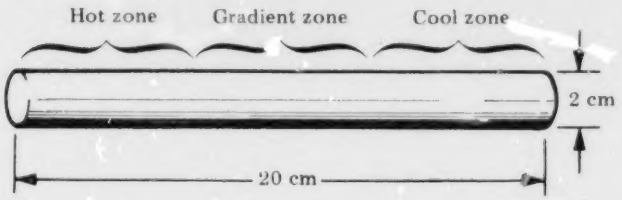


Figure 2-1 A Typical Experiment Cartridge Exposed to the Heating and Cooling Zones of the Electric Furnace

SPHERE FORMING EXPERIMENT (M553)

SCIENTIFIC OBJECTIVES

Free floating melt maximizes undercooling.

By melting metals in a weightless environment and allowing them to freeze while attached to a support or while floating free out of contact with any structure, it would be possible to demonstrate that solidification may be achieved at much lower levels of undercooling than on the Earth. It should also be possible to determine the effects of melting and solidifying in a low gravity environment on the magnetic properties of the metal, and the low gravity effects associated with freezing an alloy that has a wide freezing range and a wide density difference between components. In addition, the role of surface tension in shaping molten metal in the absence of gravity-induced forces will be studied.

MATERIAL

Microsegregation—the segregation of one element because of differences in freezing rates.

The materials selected for this experiment represent a wide range of metals. High purity nickel (Ni) was selected because it is representative of unalloyed metals having a single melting point. Nickel with one percent silver (Ni-1% Ag) was chosen because it is representative of alloys having a narrow melting range, approximately 5°C, and a density range (Ni, 8.8 gm/cm^3—Ag, 10.45 gm/cm^3) that will allow segregation upon solidification on Earth permitting relatively simple structural analysis for comparison with pure nickel. Silver in this alloy will inhibit grain growth and provide a basis for the study of microsegregation. Nickel with 30 weight percent copper (Ni-30% Cu) is an alloy with a fairly wide melting range, approximately 50°C, but almost no difference in density between the two elements. Nickel with 12 weight percent tin (Ni-12% Sn) is an alloy with wide melting range, approximately 150°C, and slight density difference between elements.

PERFORMANCE

Several samples of the four metals that are to be melted are mounted on the periphery of a wheel. An electron beam is the heat source. (See Section 4.) The sphere forming apparatus (Figure 2-2) is placed in the work chamber and the chamber is depressurized. The wheel rotates until each sample to be melted comes in line with the electron beam. *Some melted samples are ejected to float freely inside the* chamber and solidify in a spherical shape; other samples will melt and solidify while still attached to the wheel.

Sphere Forming Apparatus

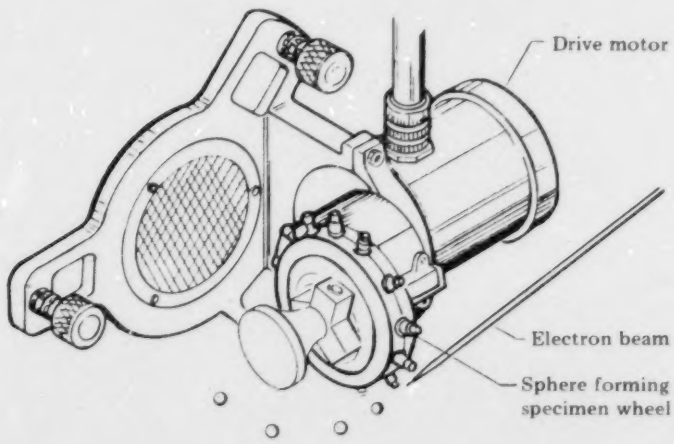


Figure 2-2 Sphere Forming Apparatus

DATA

During the experiment, the time required to melt each sample is recorded and a film recording of each melt is made. All samples are returned to Earth and the following measurements will be taken:

1) chemical analysis of the specimen by various methods such as wet chemistry, mass spectrography, vacuum fusion, and emission spectrography;
2) standard metallographic techniques to evaluate the microstructure, segregations, grain size and microporosity of specimens;
3) sphericity;
4) surface smoothness using the scanning electron microscope.

Mass Spectrography—converting molecules into ions and then separating the ions according to their mass charge ratio.

Film Records

In addition, the film records will be examined and correlated with high-speed (200 frames/sec) motion pictures taken during duplicate runs with similar samples and apparatus on Earth.

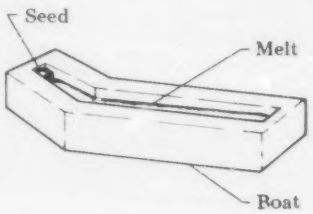
GALLIUM ARSENIDE CRYSTAL GROWTH (M555)

BACKGROUND

Single crystals of gallium arsenide (GaAs), a commercially valuable semiconductor, have been prepared by a variety of techniques: growth from the melt, growth by vapor phase reaction, and growth from metallic solutions. In all cases, the goal has been to prepare material of the highest possible chemical homogeneity and crystalline perfection.

Although growth from the melt can produce a relatively large amount of material in a given time, the technique presents a number of serious problems such as thermal convection. Crystals can be grown at temperatures lower than the melting temperature by either vapor phase or liquid solution growth techniques. In vapor phase growth, temperature fluctuations are injurious to crystal perfection, and since the system has low thermal mass, its response to system temperatures is rapid. Thus, precise temperature control of the growth apparatus is necessary. Growth of crystals from metallic solutions offers a valuable method of producing high quality material; however, thermally driven convection currents have introduced difficulties. This problem would, of course, be eliminated in weightless conditions.

In one method of controlled solidification on Earth, the molten metal is supported in a horizontal "boat" and is made to freeze progressively from one end by slowly moving an electric furnace along the boat. The leading edge of the boat is tapered to reduce the number of nuclei forming there. A small single crystal is sometimes provided as a ready-made starting point, or seed, from which the crystal can grow. The seed is oriented at the face angle of the desired crystal. The boat sometimes has a bend in it that also aids crystal orientation. (See illustration.) Then, if no competitive nucleus interferes and if there are no thermal convections, the entire melt will solidify into a single crystal.



Boat for Single Crystal Growth

MATERIAL

The material selected for this experiment is GaAs with a silicon dopant. Three arrangements of the experiments are:

1) pure GaAs crystal grown on a pure GaAs seed;
2) pure GaAs crystal grown on a silicon doped GaAs seed;
3) silicon doped GaAs crystal grown on a pure GaAs seed.

SCIENTIFIC OBJECTIVES

The major advantages anticipated from a reduced gravity environment on the growth of single crystals are:

1) improved crystalline perfection because of static convectionless melt;

2) better doping uniformity because of the absence of temperature fluctuations;

Doping—the process of adding dopant to a material

3) more uniform starting melts because the less dense solid material will not concentrate and "float";

4) more uniform growth due to static, diffusion controlled, transport of the components of the melt.

PERFORMANCE

Spherical Crystal Growth

In space the "boat" must consist of an enclosed tube. (See Figure 2-3.) The furnace is a heater in the Material Processing Facility. (See Section 4.)

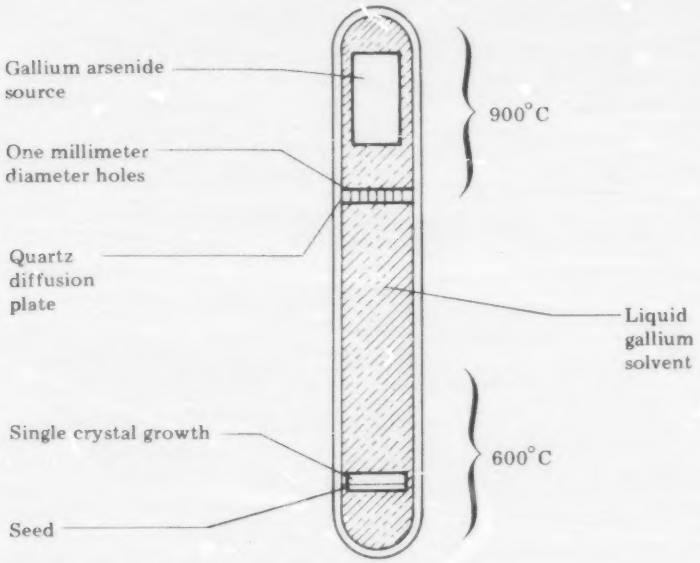


Figure 2-3 Cartridge for Spherical Crystal Growth

Upon heating, the GaAs source material will dissolve into the liquid gallium, diffuse through the diffusion plate, and disperse throughout the liquid. At the lower temperature end, the GaAs will begin its crystalline growth on a suspended single crystal seed. After the crystal growth, the temperature of the specimen must be maintained above 30° C to keep the gallium molten since it expands upon freezing. The liquid phase will be maintained until the single crystal is retrieved on Earth.

DATA

Upon return to Earth, the growth material will be analyzed by techniques already developed for the evaluation of GaAs.

Such techniques include chemical etching for the study of impurity distribution, x-ray topographic analysis for the study of crystal lattice perfection, and a variety of electrical measurements to characterize the semiconducting properties of the material. These data will be used to compare the space-grown material with material grown by similar techniques on Earth, as well as with Earth-grown material from other sources.

VAPOR GROWTH OF IV-VI COMPOUNDS

BACKGROUND

Semiconductors

The numbers IV and VI refer to the main groups of elements on the periodic table of elements. The particular compounds in this experiment are germanium telluride (GeTe) and germanium selenide (GeSe). Mixed crystals of these compounds are used extensively as semiconductors; however, more efficient and increased utilization of the compound could be realized if more perfect crystals could be produced. The problem here is that perfectly uniform crystals cannot be produced on Earth because of thermal convection in the crystal growing process. These crystals are grown from the vapor phase by placing specific amounts of semiconductor compound in an enclosed quartz tube and heating until the compound vaporizes. One end of the tube is allowed to cool and the compound begins to solidify there. As the vaporized germanium and tellurium diffuse down the tube, convection causes fluctuations in temperature. Turbulent flow develops in the transfer of the vapor mass down the tube resulting in a crystal growth with nonuniform distribution of elements. In the weightlessness of a space experiment, convection should be inhibited and vapor can be expected to flow uniformly and produce a uniform composition.

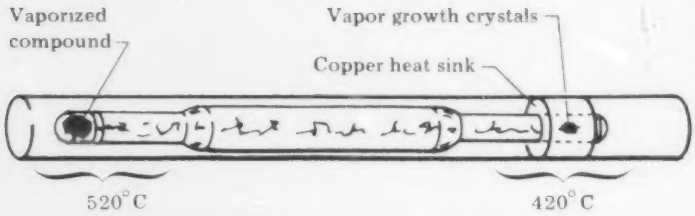
Weightlessness inhibits convection and supports uniformity of crystal growth.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to determine the degree of improvement that can be obtained in the perfection and chemical uniformity of crystals grown by chemical vapor transport under weightless conditions of space.

PERFORMANCE

Mixed crystals of GeSe and GeTe compound semiconductors will be grown by chemical transport through a temperature gradient in a transport agent, germanium tetraiodide, from polycrystalline sources of the two component materials. The growth process will be carried out in sealed quartz ampules contained in metal cartridges. (See Figure 2-4.)



The rate of vaporization is much greater in the presence of a transport agent (in this case GeI_4) than in a vacuum.

Figure 2-4 Vapor Growth of IV-VI Compounds

DATA

After return to Earth, the samples will be evaluated for chemical composition and homogeneity, crystal perfection, trace impurities, and optical and electrical properties, in comparison with samples produced under similar process conditions on Earth. The processes used for analysis are emission spectroscopy, spark source mass spectrometry, conductivity measurements, scanning electron microscope, and x-ray topography.

IMMISCIBLE ALLOY COMPOSITIONS (M557)

BACKGROUND

New Alloys

Immiscible alloys are mixtures of metals that segregate on Earth when melted. The segregation is such that the metals actually stratify; the less dense metal floats on top of the dense metal. If some combinations of these metals could be made to mix, new alloys of superior properties may be developed. For example, gold and germanium could be used as a new superconductor material. A mixture of lead, indium, and tin has a potential use as a superior superconductor in high magnetic fields.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to melt three immiscible mixtures and refreeze them to determine the effects of weightlessness on segregation.

Emission Spectroscopy—radiation emitted in the form of discrete wave lengths called spectral lines provides analytical tool.

PERFORMANCE

The experiment consists of three mixtures in three separate sections of the same experiment cartridge (Figure 2-5.) Two mixtures in the hot zone of the furnace will undergo melting

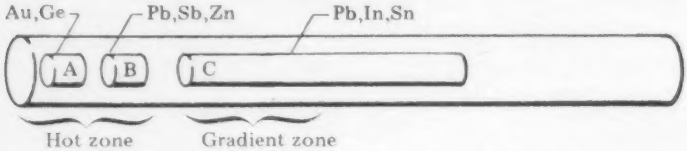


Figure 2-5 Experiment Cartridge with Ampules of Immiscible Alloys

and then isothermal solidification. The mixture in the gradient temperature zone of the experiment cartridge will be partially melted and allowed to solidify directionally.

Isothermal—indicating constant temperatures.

The immiscible alloy compositions are:

- ampule A—gold-23.2% germanium (isothermal solidification)
- ampule B—lead-45% zinc-10% antimony (isothermal solidification)
- ampule C—lead-15% indium-15% tin (directional solidification)

Densities (gm/cm^3)

- gold — 18.88
- germanium — 5.49
- lead — 11.34
- zinc — 6.92
- antimony — 6.62
- indium — 7.3
- tin — 7.28

Directional Solidification—the process of cooling a melt by drawing the heat from just one end of the container and allowing the melt to solidify toward the warmer end.

DATA

Metallurgical Analysis

Metallurgical analysis will be made on the specimens on Earth. The surfaces will be etched and viewed through a scanning electron microscope to verify the homogeneity of the mixtures.

RADIOACTIVE TRACER DIFFUSION (M558)

BACKGROUND

Diffusion in Pure Metals

In a metal bar composed of two different metals mechanically joined together, the atoms of one metal will diffuse into the other when the metals are melted; the metal with the lower melting point will diffuse faster than the other. In a pure elementary material where there is no high or low concentration of atoms and the energy level of all of the atoms is about the same, diffusion still takes place. If the bar is heated, diffusion takes place at a faster rate because excited atoms have a greater probability of changing places. Also, dislocations and vacancies are more numerous in heated metals and the atoms will tend to migrate into the holes. In a weightless condition, the extent of the diffusion should be limited to that caused by the energetic motion of the excited atoms (Brownian motion) and that caused by the artificial gravity pressures caused by spacecraft accelerations.

To measure this diffusion process, a bar of metal is used in which a certain portion contains a specific amount of radioactive isotope of the same metal. As the radioactive material diffuses, the extent of diffusion can be measured by the distribution of radiation levels. This is usually determined by measuring radiation levels of each set of lathe turnings obtained by machining the chilled bar.

SCIENTIFIC OBJECTIVE

Self-diffusion and Impurity Diffusion

The objective of this experiment is to measure self-diffusion and impurity diffusion effects in liquid metals in a weightless environment and characterize the distributing effects, if any, caused by spacecraft acceleration.

MATERIAL AND PERFORMANCE

Three rods of zinc metal are prepared with a section of radioactive zinc (Zn_{65}) plated to one end of each of two rods and in the midsection of the third rod. The zinc rods are encased in a tube of tantalum and sealed in the experiment cartridges. (See Figure 2-6.) The rods will be melted in the multipurpose electric furnace, held at a constant temperature while the radioactive atoms diffuse into the liquid metal, and then frozen.

Tracer—a radioactive isotope that can be traced through the process.

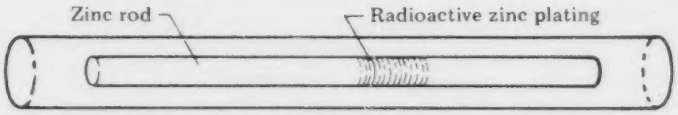


Figure 2-6 Radioactive Tracer Diffusion Experiment

DATA

Upon return to Earth, the zinc rods will be sectioned and machined, and the concentration of the diffused radioactive zinc will be measured. Results will be analyzed to determine values of the liquid diffusion coefficients in the sample materials and characterize deviations from ideal behavior because of spacecraft accelerations.

MICROSEGREGATION IN GERMANIUM (M559)

BACKGROUND

As crystals solidify, there is some segregation of impurities on a very small scale. Impurities are preferentially rejected or incorporated into a freezing crystal. Utilizing even the most careful techniques, this segregation causes nonuniformity in semiconductor "doping".

The central portion of a dendritic crystal is richer in the element having the higher freezing point. The other element,

still in the liquid phase, is forced outside the dendrite. This amount of segregation has little effect on large scale electrical characteristics because the various concentrations of different materials average out. As smaller and smaller segments of the material are used (as in microminiature circuitry), the concentration of material from one "micropoint" to the next becomes critical and "averaging" is no longer possible. In addition to this problem, gravity-induced convection within the melt further enhances the uneven distribution of materials.

Microsegregation can influence properties of crystals.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to determine the extent of microsegregation of doping material in germanium caused by convectionless directional solidification in space.

PERFORMANCE

Microsegregation

Single-crystal rods of germanium doped with antimony (an electron donor), gallium, and boron (electron acceptors) will be placed in cartridges and positioned in the multipurpose furnace so that one end of each rod extends out of the furnace hot zone. (See Figure 2-7.) When the furnace is heated, only the part of each rod that is within the hot zone will melt, leaving a solid part to serve as a seed for regrowth of the crystal. The rods will be solidified directionally at the lowest available cooling rate to promote formation of single crystals.

Seed—a single crystal used as a starting point for further crystal growth in a liquid metal.

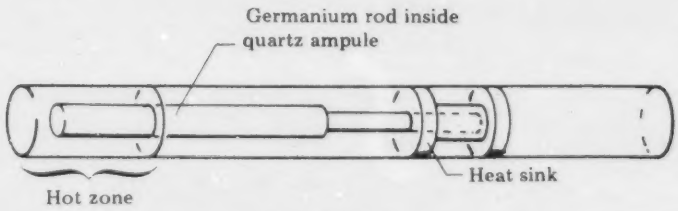


Figure 2-7 Microsegregation in Germanium

DATA

The specimens will be analyzed upon return to Earth. Concentrations of the doping impurities will be determined by electrical measurement and etching of the samples. The resulting concentration profiles will be analyzed to determine the extent of segregation of the impurities, and the effectiveness of free fall in suppressing microsegregation will be evaluated. Comparisons will be made with specimens obtained from similar experiments performed on Earth.

GROWTH OF SPHERICAL CRYSTALS (M560)

BACKGROUND

On the Earth, gravitational forces necessitate the use of a container to hold a liquid metal. Unfortunately, it is difficult to produce a metal structure with high chemical uniformity in a container, as a container material tends to alloy with the molten metal and contaminate the melt or damage the surfaces. Also, thermal convections through the liquid metal cause varying crystal growth patterns resulting in nonuniform compositions. In the weightless environment of Skylab, the metal structure can be produced without any of these effects.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to grow a doped indium antimonide crystal of high chemical homogeneity.

PERFORMANCE

Prepared samples of indium antimonide (InSb) doped with selenium will be melted inside a spherical shape quartz tube (Figure 2-8.) A solid seed crystal fixed to the inside wall of the tube will provide a starting point for the metal to crystallize. Under the influence of surface tension, the liquid metal should assume a spherical shape and attach itself to the seed crystal. Heat will be removed through the seed crystal. In this way, the crystal will grow from the inside out.

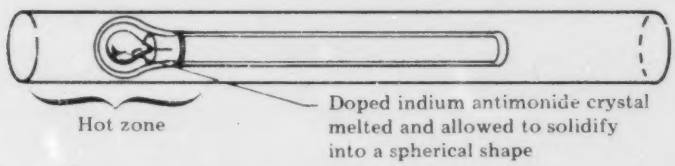


Figure 2-8 Cartridge for Spherical Crystal Growth

DATA

The sample sphere will be analyzed on Earth. The return samples will be evaluated by:

1) high resolution etching to determine chemical homogeneity (dopant concentrations). This will indicate growth interface stability;
2) x-ray diffraction to characterize structural perfection;
3) measurements of electrical properties, e.g., carrier concentration, mobility, and lifetime;
4) scanning electron microscope and ion microprobe analysis to determine homogeneity of samples on an even smaller scale, and the topography and surface roughness.

WHISKER—REINFORCED COMPOSITES (M561)

BACKGROUND

High Strength Whiskers

The bond between atoms in a metal is very strong, much stronger than any practical material that has been produced. However, metals are not as strong as they could be because of defects that exist in the crystal. A displaced atom, a void, a misaligned crystal, and an impurity are examples of defects that cause stress fields and result in a weaker metal. Crystals of practical size cannot be produced free from defects. However, the theoretical high strength can be approximated in "whiskers." Whiskers are small crystals that are produced so that very few defects can occur. These small whiskers can be used as reinforcing material for other metals. If these high strength whiskers are distributed throughout a material of relatively low strength, the resulting alloy could approach the strength of the whiskers. The total structure then absorbs a loading stress that would easily break the weaker element.

The silicon carbide (SiC) whisker has very high strength and can be used to mix with various metals. However, good quality composite metals reinforced with SiC whiskers have been difficult to produce because the low density whisker tends to float out of the high density molten metal. Another problem is that the metal does not "wet" the SiC whisker (i.e., ideal contact with the whisker is not achieved) and voids occur.

Sintered—a process for forming metal pieces by compacting powdered metal.

In this experiment SiC whiskers will be oriented lengthwise in a sintered pack of silver. The weightless environment in the Skylab is expected to improve the uniformity of distribution of the metal and the whiskers since density differences will not be a factor. A compressive force will be externally applied continuously during storage, melting, and solidification to remove voids that may occur.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to produce void-free samples of silver uniformly reinforced with oriented SiC whiskers.

PERFORMANCE

Sintered compacts of silver containing distributions of unidirectionally oriented SiC whiskers (1 mm long by 1 micron diameter) will be melted in the multipurpose electric furnace under pressure from a piston actuated by a spring. (See Figure 2-9.)

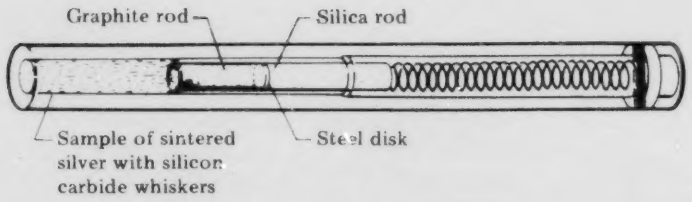


Figure 2-9 Whisker-Reinforced Composites

DATA

After solidification and return to Earth, the samples will be evaluated as follows:

1) calculated ideal density compared with the measured density;
2) mechanical tests conducted to determine Young's modulus and hardness numbers;
3) x-ray microanalysis performed and the internal structure of the sample checked with electron microscope to verify a void-free homogeneous mixture.

INDIUM ANTIMONIDE CRYSTALS (M562)

BACKGROUND

Indium antimonide crystals (InSb) are the basic components of some semiconductors. In order to have the desired electrical properties, the crystals are doped with small amounts of materials such as tellurium. Growth rate fluctuations due to gravity-induced convection currents and rejection of the dopant from the growing crystal interface cause serious problems in the production of these crystals. The convection currents periodically cool and heat the interface, thus initiating faster and slower growth rates. Since the concentration of dopant depends on the growth rate, there are periodic bands of impurities in the resultant crystal. Thus electrical properties vary with the widely dispersed dopant.

Dopant concentration is more uniform in the absence of convection currents.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to produce doped semiconductor crystals of high chemical uniformity and structural perfection, and to evaluate the influence of weightlessness in attaining these properties.

PERFORMANCE

High quality single crystals of InSb will be prepared in the laboratory, doped with tellurium, precision machined, and

etched to fit into heavy wall quartz ampules. Half of each crystal (about 7.6 cm length) will be melted and regrown at a rate of 1.3 cm/hr using the unmelted half as a seed.

Ion Microprobe Analysis—an electron beam excites the x-ray spectra of the elements present in a specimen. The x-rays are analyzed by their wave lengths as the "probe" is moved along the specimen.

DATA

Upon return to Earth, 2.5 cm of each crystal will be remelted and regrown in the same ampule under the same thermal conditions that it was subjected to in space. Subsequently, extensive analysis will be carried out on all three portions of the crystals by using the following techniques:

Analytical Methods

1) high resolution etching techniques in conjunction with interference contrast microscopy for establishing dopant microdistribution and morphology of the growth interface;
2) x-ray diffraction;
3) x-ray topography;
4) scanning electron microscope;
5) microanalytical techniques—spectroscopy and ion microprobe analysis;
6) established parameters (carrier concentration and mobilities) as a function of temperatures down to liquid helium or below.

MIXED III-V CRYSTAL GROWTH (M563)

BACKGROUND

Mixture ratio control is key to electrical properties.

The numbers III-V refer to the main group of elements in the periodic table of elements. The metals in this experiment, indium antimonide and gallium antimonide, are used in the production of semiconductors. In order to produce semiconductors with optimum conduction and bandpass capabilities, it is necessary to mix the metal elements in specific proportions. The problem here is that convections in the liquid cause nonuniform crystal growth through local segregation of the materials. Uneven mixture ratios result.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to determine how weightlessness effects directional solidification of semiconductor alloys and, if single crystals are obtained, to determine how their semiconducting properties depend on alloy composition.

PERFORMANCE

Alloys of indium antimonide (InSb) and gallium antimonide (GaSb) in proportions of 0.1 indium to 0.9 gallium, 0.3

indium to 0.7 gallium, and 0.5 indium to 0.5 gallium are placed in separate silica ampules. These samples will be melted and allowed to solidify slowly.

DATA

The ingot samples will be brought back to Earth for analysis to determine microstructure and compositional uniformity by x-ray diffraction, electron microprobe analysis, and electrical measurements. Samples solidified under similar conditions on Earth will be used for comparison.

HALIDE EUTECTICS (M564)

BACKGROUND

As a liquid eutectic composition of two components solidifies, the crystal formation of one component rejects the other component resulting in a parallel growth of crystals that can take the form of either platelets or rods extending throughout the solid in the direction of crystal growth.

Eutectic—an alloy with a composition such that all elements freeze simultaneously.

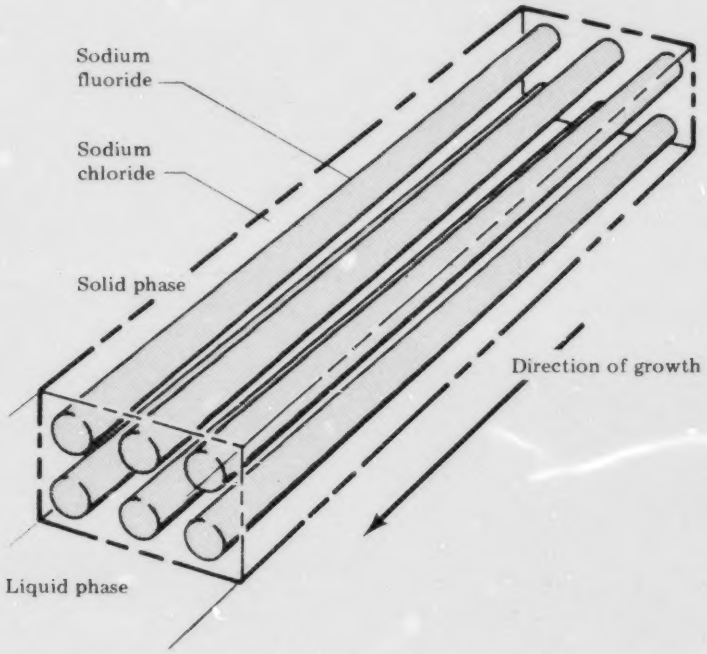


Figure 2-10 Rods in Lamaller Crystal Growth

The eutectic composition of sodium chloride and sodium fluoride solidifies into crystalline rods of sodium fluoride. The rods are transparent through their length and if a crystal of this composition could be grown without defects, it could be used as fiber optics material. The problem is that in the

formation of the crystal on Earth, thermal convection within the liquid phase causes discontinuous crystal growth. As a result, the sodium fluoride rods are too short for any practical use. In a weightless environment, the thermal convection within the melt will not exist and a fiberlike crystal should result.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to produce highly continuous controlled structures of halide compositions.

PERFORMANCE

Ingots of the sodium fluoride and sodium chloride eutectics 1.27 cm in diameter and about 10 cm long, will be grown by melting the mixture and allowing them to cool slowly and directionally solidify.

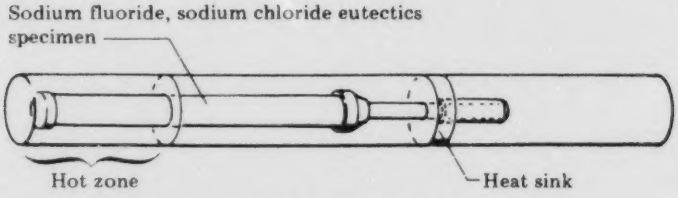


Figure 2-11 Experiment Cartridge for Halide Eutectics

DATA

Postflight analysis will include:

1) metallographic examination and x-ray diffraction study to identify and characterize alloy phases;
2) electron microprobe analysis to determine concentration in the transverse and longitudinal sections;
3) measurement of electrical, superconducting, and optical properties.

SILVER GRIDS MELTED IN SPACE (M565)

BACKGROUND

Cohesive forces become dominant forces in orbital environment.

The forces of gravity on a liquid metal are much greater than the forces of cohesion. Consequently, a solid metal structure on Earth loses its shape when melted. In a weightless condition, a liquid metal would be subject to surface tension forces only, which would tend to draw the melt into a spherical shape.

By using a specific configuration for the original material, or by applying physical constraints to the specimen, other

shapes might be achieved. In this experiment, discs with various patterns of holes are used to determine how the surface tension forces reconfigure the patterns. The resulting discs will have shapes formed by surface tension forces. Since unrestricted surface tension tends to minimize the surface area of a given configuration, the resultant shapes will be important considerations in the design of grids with optimum strength-to-weight ratios.

Surface tension forces may lead to optimal strength/weight ratios.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to determine how pore sizes and shapes change in grids when they are melted and resolidified in a zero gravity environment.

PERFORMANCE

Discs of silver with small holes of different number and spacings will be mounted inside a quartz ampule and melted and refrozen in space.

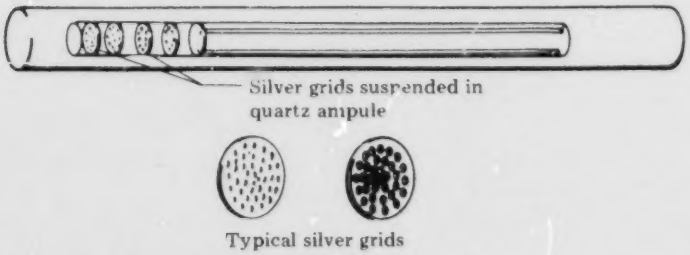


Figure 2-12 Silver Grids Melted in Space

DATA

The samples returned from space will be studied to characterize the morphology and geometry of the configurations.

ALUMINUM—COPPER EUTECTIC (M566)

BACKGROUND

Lamellar Structures

The aluminum and copper eutectic composition is representative of types of alloys that freeze in a lamellar crystal pattern, i.e., forming platelets. (See Figure 2-13.) However, thermal convections within the melt occurring during the solidification process can cause irregular platelet growth. The weightless environment of Skylab will eliminate the problem of gravity-induced thermal convection and the crystals should grow in a continuous lamellar pattern. If continuously perfect lamellar structures are formed, various alloys with lamellar growth patterns could be grown with

numerous practical applications such as superconductors, optical material, capacitors, semiconductors, or directional magnetics.

SCIENTIFIC OBJECTIVE

The objective of this experiment is to determine the effects of weightlessness on the formation of lamellar structure in eutectic alloys when directionally solidified.

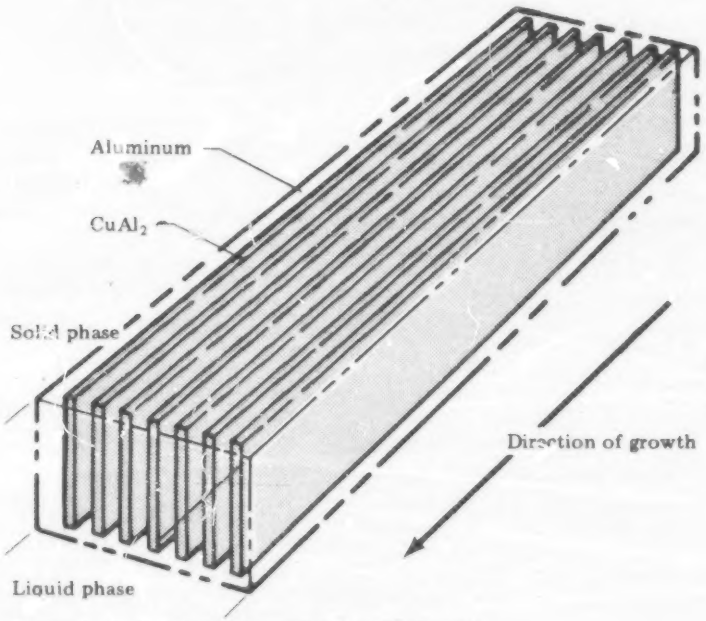


Figure 2-13 Platelets in Lamaller Crystal Growth

PERFORMANCE

Three rods of 67% pure aluminum—33% copper eutectic alloy in individual ampules will be partially melted and then directionally solidified.

DATA

The returned samples will be evaluated by comparisons with samples produced under similar conditions on the Earth.

CLASSROOM DEMONSTRATIONS

DIFFUSION WITH RADIOACTIVE TRACERS

Relatively safe radio isotopes such as iodine 127 may be used to determine diffusion rates in solution.

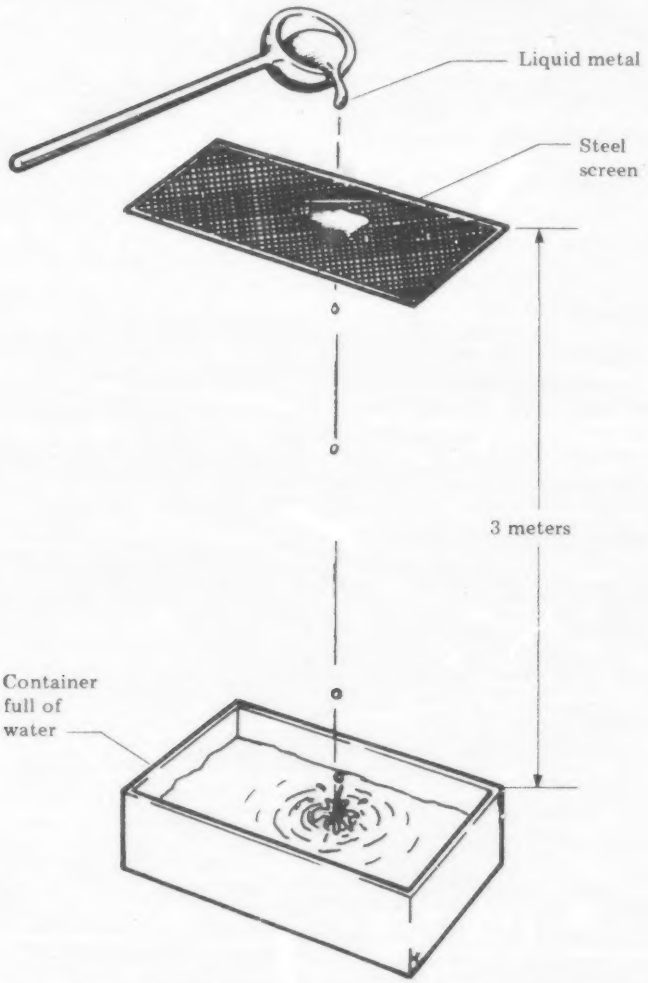
Student Activity

Fill a tube with KI or NaI solution of known concentration. Place the tube in a horizontal position. Slowly introduce a

known quantity of radioactive iodine at one end. Periodically remove the cover and sample a small amount of the solution at incremental distances from the end. Repeat the experiment with vertical tubes of the solution and add the radioactive iodine to the top of the tubes. Check for diffusion downward.

LIQUID METAL IN FREE FALL

The shape of metal in a weightless environment can be demonstrated by allowing liquid lead or tin to fall some distance (over 3 meters) into a tank of water. Melt some lead or tin and carefully pour the liquid over a heated steel screen. The molten metal will pass through the screen and drops will fall into the water. (See Figure 2-14.) The metal will freeze quickly thereby retaining most of the free-fall shape.



Sphere forming experiment

Figure 2-14 Demonstration of Liquid Metal in Free Fall

Wide Range of Drop Shapes

The solidified drops of metal may have other than spherical shapes because of various stresses that exist in the experiment. The liquid metal may develop oscillations resulting from the stretching and release of the droplet. Also, the drag forces of the fall through air will tend to flatten the drop. Try dropping the liquid metal from varying heights up to several feet and note the variations in drop shape for the different increments in drop height.

CAUTION **Adequate safety precautions should be applied to avoid burns.**

CRYSTALS OF SODIUM BROMATE

Dissolve a saturated solution of sodium bromate in water and allow the liquid to cool. Saturate a string with sodium bromate crystals and allow to dry. Suspend the string in the solution and place the apparatus in a refrigerator. Cool the solution to about 10 to 20°C for three or four days. Crystals of sodium bromate will attach and grow seed crystals on the string.

Section 3

Space Operations Support Equipment

The three experiments described in this section relate primarily to processes and techniques having applications in future manned space flight operations. In addition, the experiments produce data that will enhance the understanding of the physical principles involved in particular processes.

ZERO-GRAVITY FLAMMABILITY (M479)

BACKGROUND

Flame Propagation Influenced by Gravity

In the Earth environment and in the absence of artifically supplied oxidizers, convection of air plays an important part in the burning of materials. The amount of oxygen provided to a fire depends on convection of air and it is convection-induced heat flow that causes material to burn faster from the bottom up than from the top down.

In weightlessness, convection currents are diminished or even eliminated. The burning of materials in weightlessness permits the study of diffusion and other influences in the burning process.

Flame Propagation—process in which the flame migrates.

The experiment will be conducted in the work chamber described in Section 4.

SCIENTIFIC OBJECTIVES

The objectives of this experiment are to determine the:

1) extent of surface flame propagation and flashover to adjacent materials;

2) rates of surface and bulk flame propagation;

3) ability of the flame to extinguish itself;

4) ability of vacuum to extinguish the flame;

5) ability of water spray to extinguish the flame.

MATERIALS

The materials selected for this experiment are aluminized mylar film, nylon sheet, neoprene coated nylon fabric, polyurethane foam, bleached cellulose paper, and Teflon fabric.

In six tests, the samples of the six materials will be ignited and then extinguished by opening the work chamber to the vacuum of space. In another six tests on the same materials, water will be used to extinguish the fire. (See Figure 3-1.)

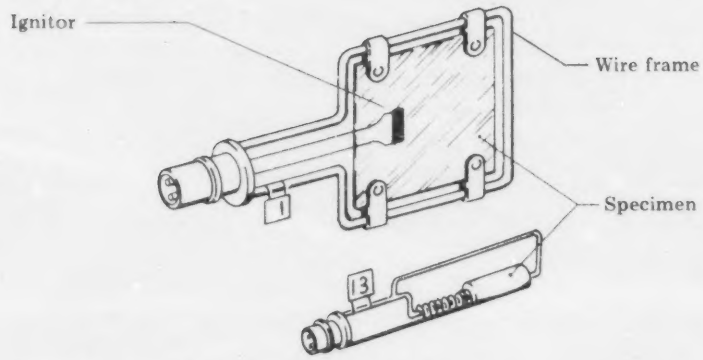


Figure 3-1 Typical Flammability Specimens

Another set of tests using bleached cellulose paper will investigate the capability of a flame to flash over from one material to another across different distances. Two sheets of the material are mounted in frames with separation distances of 0.3, 0.6, and 1.27 cm.

DATA

Motion picture coverage of ignition and flame propagation will serve as the primary data record. Color film coverage at 24 frames per second is used for all but one test series; that test will use infrared film. Each test will be photographed in its entirety so that combustion rates can be determined by postflight analysis of the film.

METALS MELTING EXPERIMENT (M551)

BACKGROUND

This experiment will melt the surface of a metal during free-fall conditions. Solidification processes in metals are expected to behave differently under weightless conditions than under conditions on Earth. In the absence of gravity, heat transport should be dominated by conduction, and mass transfer should be dominated by diffusion. On Earth, both of these processes are heavily influenced by gravity-induced circulating convection currents.

An electron beam will be used as the heat source. (See Section 4.) Analyses of these specimens will provide new data relative to metal flow and hardening. The migration and diffusion of the melted and frozen metals will be studied. The data obtained will relate to the properties of weld strengths and control of the welding process.

SCIENTIFIC OBJECTIVE

One objective of this experiment is to demonstrate metal joining and cutting techniques and define their tolerances and

limitations in a space environment. A second objective is to analyze the behavior and solidification of molten metal in reduced gravity.

MATERIALS

The materials for this experiment support this objective in that a wide range of phenomena can be studied using the following samples:

1) aluminum 2219, which represents a solid solution with components of different density, has been studied extensively, especially with respect to dendritic growth;

2) stainless steel 304 represents an alloy with components of similar density;

3) tantalum represents a pure metal and its solidification behavior differs substantially from that of the other two alloys.

Aluminum 2219 is an aluminum-copper alloy that has superior properties to other aluminum alloys; it has excellent weldability and is used extensively in the construction of airborne and space vehicles.

Stainless steel 304 is low carbon steel used for food processing equipment, chemical equipment, recording wire, and structural applications.

PERFORMANCE

Weld Test

Basically, the experiment will be conducted like a conventional weld test. Discs of the sample metals will rotate in front of an electron beam heat source (Figure 3-2). The electron beam is powered at about 1.6 kw and focused on a spot about 1.5 mm in diameter on the sample disc. As the disc moves, the melt left in the track will freeze rapidly because the rest of the disc serves as a heat sink. Each disc is machined to produce several thicknesses of metal. At the end of one revolution of the disc the beam will heat one spot until a hole is burned through the metal.

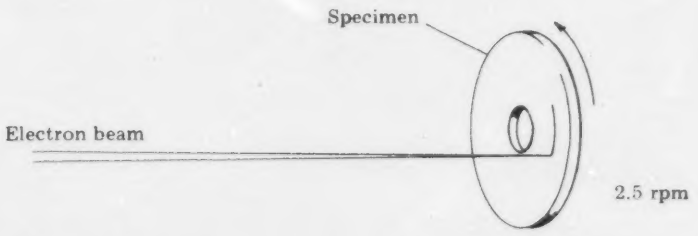


Figure 3-2 Materials Melting Experiment

DATA

The operator will record process conditions and note observations. Color motion pictures will be taken at a rate of 24 frames/sec during each run.

Analytical Methods

After the specimens are returned to Earth, each sample will be analyzed using various techniques including visual examination, radiography, physical testing, materials analysis, scanning electron microscopy, and x-ray diffraction.

The film records will be examined and correlated with high-speed (240 frames/sec) motion pictures taken during duplicate runs with similar samples and apparatus on Earth.

EXOTHERMIC BRAZING EXPERIMENT (M552)

BACKGROUND

Pipe joining experiment

An exothermic brazing package is a device used to repair or join a pair of metal tubes. A metal joining sleeve with braze material contained in internal annular grooves in the sleeve and the heating material are all contained in the package. The heating material is ignited and when melting occurs the braze material flows to form a seal and structural joint between the tubes and the sleeve (Figure 3-3).

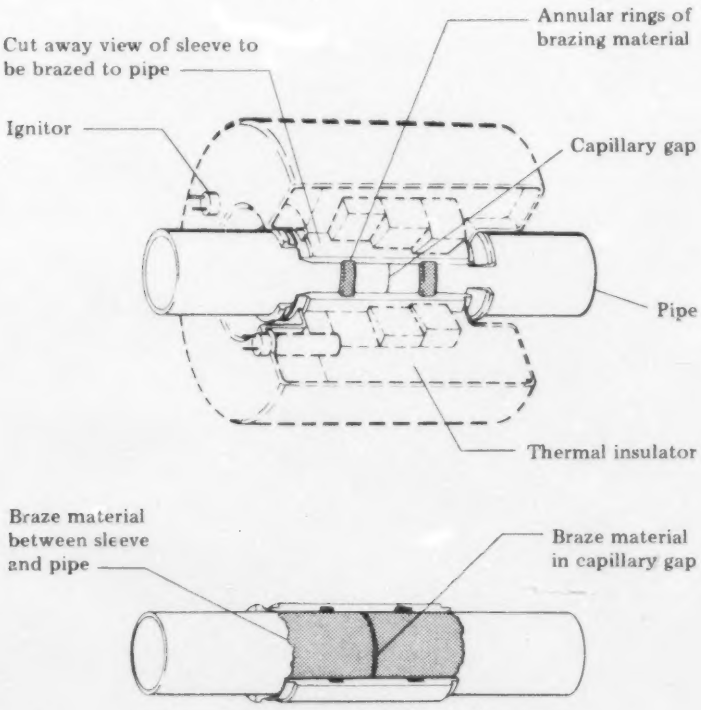

Annular rings of brazing material
Cut away view of sleeve to be brazed to pipe
Ignitor
Capillary gap
Pipe
Thermal insulator
Braze material between sleeve and pipe
Braze material in capillary gap

Figure 3-3 Exothermic Brazing Experiment

The capillary flow of the braze material and the degree of contact with the sleeve and tube metals depend greatly on the surface tension of the liquid metal and on the gap characteristics.

Flow Velocity of Liquid Metal

The equation, $V = D\gamma/6\mu L$, represents the flow velocity of liquid metal in the absence of gravity

where

D = gap distance

γ = surface tension in the liquid

μ = liquid viscosity

L = distance from the liquid to the capillary opening,

suggesting very high velocities may be developed in a wide gap capillary. The velocities might be such that turbulent flow takes place. (This will be of interest since turbulent flow in this application has never before been studied.)

Experiments show that this equipment can be used in very low ambient pressure, which suggests a potential application for construction and repair in future space programs.

SCIENTIFIC OBJECTIVES

The objectives of this experiment are to:

1) test and demonstrate this method of brazing in space repair;

2) study surface wetting and capillary flow effects in weightless molten metals;

3) study concentration gradients at the braze base metal interface resulting from diffusion and convection of base metal into the liquid during and after capillary flow, as well as diffusional transport of copper into the solid base metal (silver being substantially insoluble in both 304L steel and pure nickel).

EQUIPMENT

The experiment equipment consists of a sample tube with a gap cut in a portion of the center representing two tubes end to end (Figure 3-3). Four samples of different configurations of metal or gap dimensions are used. They are:

1) 304L stainless steel with a 0.127-mm capillary gap;

304L stainless steel contains a maximum of 0.03% carbon.

2) 304L stainless steel with a 0.5-mm capillary gap;

3) pure nickel with a 0.25-mm capillary gap;

4) pure nickel with a tapered gap ranging from 0.75 mm to zero.

304L stainless steel was selected to simplify sample analysis while retaining a material of engineering interest. It is superior to other stainless steels in terms of cleanliness and weldability.

Pure nickel was selected since this material is readily wet (virtually zero contact angle) by the eutectic braze alloy at any vacuum greater than 10^{-3} torr.

Torr—a unit of pressure, 1 mm of Hg

Braze alloy, a eutectic composition of 71.8% silver, 28% copper, and 0.2% lithium, is included in a sleeve that fits over the sample inside the exothermic heater.

Radioactive tracer is added to more readily assess braze alloy flow and dispersion patterns.

PERFORMANCE

The brazing packages will be ignited in sequence and each will burn for 90 seconds. Gases from the brazing packages will be vented to the work chamber and from the chamber to space. After brazing tests, the case will be returned to Earth unopened for analysis.

DATA

Each sample will be analyzed and examined by radiographic, metallographic, electron microscopy, and physical test methods.

Section 4

Experimental Program Supporting Facilities

EXPERIMENTAL PROGRAM SUPPORTING FACILITIES

All of the experiments discussed in this volume are performed in the Materials Processing Facility (M512). This facility consists of an enclosed work chamber and equipment storage provisions. Figure 4-1 illustrates the major features of the facility.

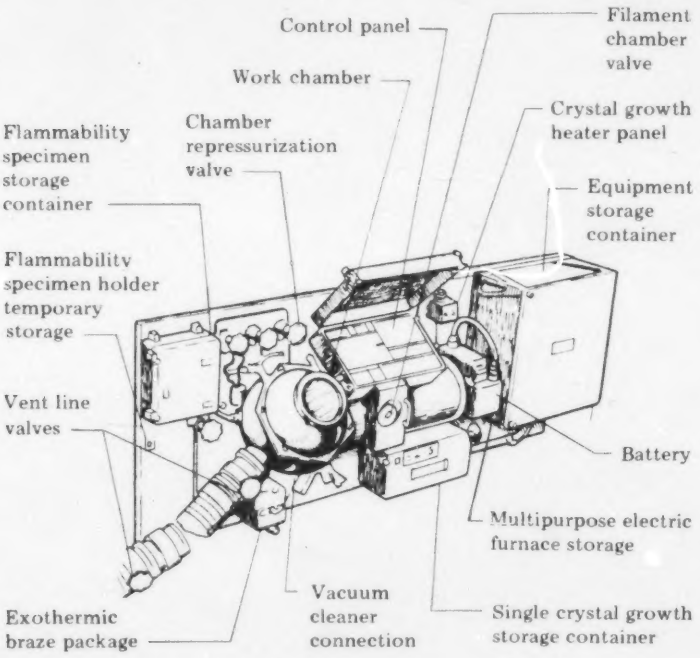


Figure 4-1 Materials Processing Facility

WORK CHAMBER

Access

The work chamber (Figure 4-1) is a 41.27 cm sphere with a hinged cover to provide access to the chamber of installation for removal of experiment equipment. The cover has a viewing port with x-ray opaque glass to protect the crew from radiation while observing the experiment process. The work chamber is connected to the space environment with a 10.16 cm line that allows the chamber to be depressurized by opening two vent valves.

Work chamber is an experimental laboratory supporting a wide range of experiments

Viewing

A second viewport is provided for the 16mm camera. A floodlight located next to the camera port illuminates the interior of the chamber. A vacuum cleaner port is provided for cleaning the work chamber after performance of experiment M479. The vacuum cleaner will also be used to retrieve the "floating" spheres formed during experiment M553.

A chamber pressurization system is provided that allows the pressures in the work chamber and spacecraft to equalize. A water system is provided to spray a series of flammability samples inside the work chamber in the zero-gravity flammability experiment (M479).

Other provisions include viewing mirrors and protective shields, and a heat sink for the crystal growth experiments.

A tungsten absorber plate is permanently mounted in the work chamber. This plate is attached to the chamber wall directly opposite the electron beam gun to protect the wall from the beam. The electron beam is the energy source for some of the experiments.

ELECTRON BEAM GUN

Supporting Equipment

In the electron beam gun electrons from a hot filament are accelerated by a high potential (20 kv) and then regulated and focused to bombard the experiment workpiece. The beam produces 1.6 kw at 80 mA. A simplified diagram of the system is shown in Figure 4-2.

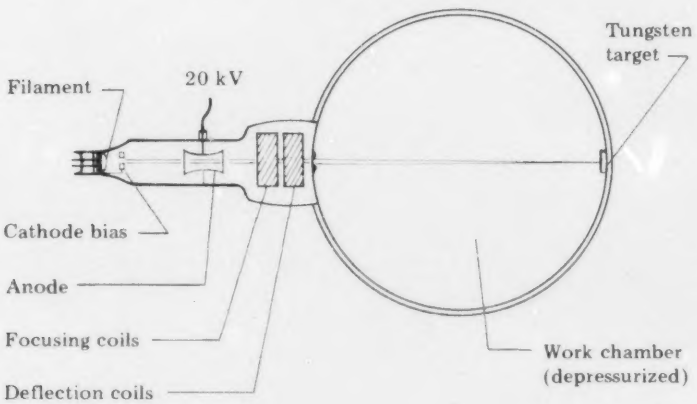


Figure 4-2 Electron Beam Gun Diagram

The filament serves as the source of the electrons which then pass through a cathode bias cup causing them to converge and pass through a hole in the center of the anode. The velocity of the electrons is a function of the potential between the cathode and anode, and the highest velocity is at the anode. A focus coil causes the electron beam to converge on the workpiece where the diameter of the electron beam is about 3 mm.

MULTIPURPOSE ELECTRIC FURNACE

The multipurpose electric furnace (Figure 4-3) is intended to supplement the capabilities of the materials processing

facility by providing means to perform numerous experiments on solidification, crystal growth, and other processes involving phase changes in materials. The furnace system will be used to perform eleven experiments involving phase changes at elevated temperatures in systems comprising selected combinations of solid, liquid, and vapor phases.

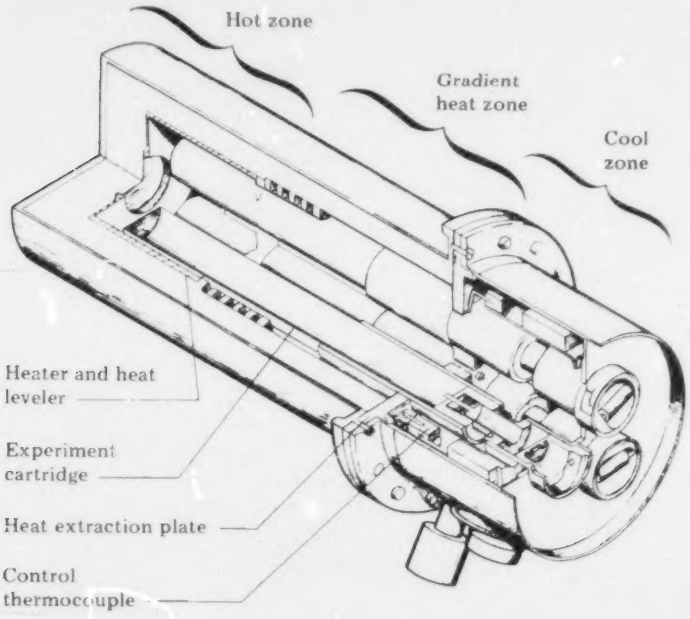


Figure 4-3 Cut-Away View of the Multipurpose Furnace

The multipurpose electric furnace has three specimen cavities which allow three samples to be processed at one time. The furnace is constructed so as to provide three different temperature zones along the length of each sample cavity.

Hot zone (at the end of the sample cavity)—constant temperature up to 1000°C can be attained

Each sample material will be enclosed in a cartridge which is designed to provide temperature distribution through use of various insulating and conducting materials.

Gradient zone (next to the hot zone)—temperature gradients ranging from 20°C/cm to 200°C/cm can be established in samples

Experiment Control Pre-programmed

A control system is provided to control the furnace temperature. Any specified temperature within the furnace's capability (0 to 1000°C) can be selected by the astronaut operating the system. Timing circuits in the controller will enable the astronaut to program the length of time spent at a particular temperature and the cooling rate of the furnace. Active temperature control will continue during programmed cooling.

Cool zone—heat conducted along samples is rejected by radiation to a conducting path that carries the heat out of the system

Section 5

Appendixes

APPENDIX A

GLOSSARY

GLOSSARY

Ampule	An enclosed glass or quartz tube.
Defect	A void, vacancy, dislocation, or an impurity within the crystal structure.
Dendrite	A tree-like crystal formed during solidification of metals.
Dendritic Growth	The second state of crystal growth as dendrites grow larger.
Directional Solidification	The process of cooling a melt by drawing the heat from just one end of the container and allowing the melt to solidify toward the warmer end. This is usually done to produce large elongated crystals or single crystals.
Dopant	An impurity added to semiconductor crystals as a donor or acceptor of free electrons.
Doping	The process of adding dopant to a material.
Etching	The process used to reveal the boundaries between crystals. Crystal boundaries are much too narrow to be visible under a microscope. A polished metallic surface is immersed in a weak acidic or basic solution that attacks the surface at a rate which varies with the crystalline orientation. This produces a plateau effect that can be seen with shadows produced with proper lighting.
Emission Spectrography	Analytical spectrometric methods utilize the characteristic radiation produced when materials are subjected to thermal or electrical sources. These sources excite the atoms or molecules to energy levels above the ground state. As they return from higher energy states, radiation is emitted in the form of discrete wave lengths called spectral lines.
Eutectic	Generally refers to the composition of an alloy such that all elements within the alloy freeze at the same temperature. The temperature at that freezing point is referred to as the eutectic temperature.
Fiber Optics	A field of optics that utilizes translucent or transparent fibers to conduct light along their length. Light rays enter the fiber through one end and are reflected internally along walls of the fibers and finally emerge from the other end. The fibers are usually flexible or they can be formed in a bend enabling the light to be ducted around corners.
Grain	A single crystal grown to the boundaries of other crystals that are oriented in a different direction.
Halides	A compound of a halogen with another element.

Impurity	A small part of one chemical substance dissolved in a metal. It is usually an unwanted substance although dopant is sometimes referred to as an impurity.
Isothermal Solidification	The process of cooling a melt uniformly throughout.
Lamellar	Refers to the parallel crystal growth of two different components of a eutectic composition.
Mass Spectrography	The technique for converting molecules into ions and then separating the ions according to their mass charge ratio. The record of the mass distribution and relative abundance of ionic products is the mass spectrum that is used in determining the molecular structure.
Microporosity	Holes or cavities that result from gases entrapped in the freezing metal, or internal shrinkage of metal that can occur while freezing, or the vacancies left by a group of atoms diffusing from one metal into an adjoining metal.
Microprobe Analysis	An analysis of the chemical makeup of a specimen of metal. An electron beam is focused on the specimen exciting the x-ray spectra of the elements present in the specimen. The x-rays are then analyzed by their wavelengths as the "probe" is moved along the specimen. Particular wavelengths correspond to elements within the specimen.
Microsegregation	The segregation of one element because of differences in freezing rates. As dendritic growth begins in one element, the other element still in the liquid phase, is forced outside the dendrite.
Morphology	The study of structure and form.
Nucleation	The formation of small nuclei crystals in a metal in the liquid phase as it begins to cool.
Nuclei or Nuclides	A group of atoms that have lost thermal energy and interatomic attraction has pulled them together in a definite crystalline cluster.
Phase	The energy state of the metal. It is either in the solid phase, liquid phase, vapor phase, or combinations of these.
Seed	A term used to describe a single crystal used as a starting point for further crystal growth in a liquid metal.
Semiconductor	A metal used as an electrical conductor whose range of resistivity lies between good conductors, such as silver and gold, and insulators.
Sintered	A process of compacting a metal. Usually the metal is in a powdered from and then compressed. The compact is heated to a temperature below the melting point.

Thermal Etching	The surfaces of some metals change shape when held at high temperatures for long times. The shape changes to form a "grove" wherever the crystal boundary meets the surface.
Tracer	A material of some kind added to another material enabling observation to be made of the movement of particles within the material. In metals, a radioactive source or a dye is used.
Undercooling	The cooling of a liquid metal below its freezing point to start the nucleation process.
Unit Cell	The smallest group of atoms processing the symmetry of the crystal which, when repeated in all directions, will develop the crystal lattice.
Void	A vacancy in the crystal structure.
Weightlessness	The state of being without weight.
Whiskers	Elongated single crystals grown under controlled conditions to minimize the possibility of defects.
X-Ray Diffraction	Crystals are symmetrical arrays of atoms containing rows and planes of high atomic density. Because of this orderly arrangement, the crystals act as a three-dimensional diffraction grating. Low voltage x-rays (20-50 kilovolts) have wavelengths of proper magnitude to be diffracted by crystals. The patterns produced by the image of reflected x-rays contains sufficient information to determine both the dimensions of the unit cell of the crystal lattice and the atomic arrangement within the cell. Qualitative identification of structures can be made by comparison of the interplanar spacing values of the specimen pattern with an index of standard patterns.

APPENDIX B

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BIBLIOGRAPHY

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